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MIKHAIL KEJZELMAN et al.

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Sir:

The benefit of the filing date of the following priority foreign application(s) in the following foreign country is hereby requested, and the right of priority provided in 35 U.S.C. § 119 is hereby claimed.

Country: Sweden

Patent Application No(s).: 0203134-2

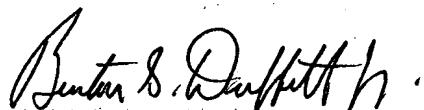
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In support of this claim, enclosed is a certified copy(ies) of said foreign application(s). Said prior foreign application(s) is referred to in the oath or declaration. Acknowledgment of receipt of the certified copy(ies) is requested.

Respectfully submitted,

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METHOD OF PREPARING IRON-BASED COMPONENTS

FIELD OF THE INVENTION

The present invention relates to metal powder compositions useful within the powder metallurgical industry. More specifically the invention concerns a method for the preparation of components having high density by using these compositions.

There are several advantages by using powder metallurgical methods for producing structural parts compared with conventional machining processes of full dense steel. Thus, the energy consumption is much lower and the material utilisation is much higher. Another important factor in favour of the powder metallurgical route is that components with net shape or near net shape can be produced directly after the sintering process without costly shaping processes such as turning, milling, boring or grinding. However, normally a full dense steel material has superior mechanical values compared with PM components. This is mainly due to the occurrence of porosity in the PM components. Therefore, the strive has been to increase the density of PM components in order to reach values as close as possible to the density value of a full dense steel.

Among the methods used in order to reach higher density of PM components the powder forging process has the advantage that full dense components may be obtained. The process is however costly and is utilised mainly for mass production of heavier components, such as connection rods. Full dense materials can also be obtained by elevating the pressure, such as in hot isostatic pressing, HIP, but also this method is costly.

By using warm compaction, a process where the compaction is performed at an elevated temperature, typically at 120 to 250 °C, the density can be increased with about 0,2 g/cm³, which results in a considerable improvement of the mechanical properties. A disadvantage is however that the warm compaction method involves additional investment and processing. Other processes, such as double pressing, double sintering, sintering at elevated temperatures etc, may further increase the density. Also these methods will add further production costs hence reducing the overall cost effectiveness.

In order to expand the market for powder metallurgical components and utilise the advantages with the powder metallurgical technique there is thus a need for a simple, less expensive method of achieving high density compacts with improved static and dynamic mechanical strength.

Summary of the invention

It has now been found that high density components can be obtained by using high compaction pressures in combination with coarse powders. In view of the general knowledge, that conventionally used powders, i.e. powders including fine particles, cannot be compacted to high densities without problems with e.g. damaged or deteriorated surfaces of the compacts this finding is quite unexpected. Specifically, the method according to the present invention includes the steps of providing an iron-based powder essentially free from fine particles; optionally mixing said powder with graphite and other additives; uniaxially compacting the powder in a die at high pressure and ejecting the green body, which may subsequently be sintered.

Detailed description of the invention

The term "high density" is intended to mean compacts having a density of about at least 7.3 g/cm³. Components having lower densities can of course also be produced but are believed to be of less interest.

The iron-based powder according to the present invention includes pure iron powder such as atomised iron powder, sponge iron powder, reduced iron powder; partially diffusion-alloyed steel powder, and completely alloyed steel powder. The partially diffusion-alloyed steel powder is preferably a steel powder alloyed partially with one or more of Cu, Ni, and Mo. The completely alloyed steel powder is preferably a steel powder alloyed with Mn, Cu, Ni, Cr, Mo, V, Co, W, Nb, Ti, Al, P, S and B. Also stainless steel powders are of interest.

As regards the particle shape it is preferred that the particles have an irregular form as is obtained by water atomisation. Also sponge iron powders have irregularly shaped particles and are of particular interest.

A critical feature of the invention is that the powder used have coarse particles i.e. the powder is essentially without fine particles. The term "essentially without fine particles" is intended to mean that less than about 5 % of the powder particles have a size below 45 µm as measured by the method described in SS-EN 24 497. So far the most interesting results have been achieved with powders essentially consisting of particles above about 106 µm and particularly above about 212 µm. The term "essentially consists" is intended to mean that at least 10 %, preferably at least 40 %, most preferably at least 60 % of the particles have a particle size above 106 and 212 µm, respectively. So far the best results have been obtained with powders having a particle size above about 212 µm and only less than 5 % below 212 µm. The maximum particle size may be about 2 mm.

The particle size distribution for iron-based powders used at PM manufacturing is normally distributed with a gaussian distribution with a median particle diameter in the region of 30 to 100 µm. A certain content of finer particles is recommended as these particles acts as so called "sintering aids". Metals Handbook, ninth ed., vol 7, p 317, teaches that " , a smaller grain size (or smaller particle size) aids sintering densification and final properties."

Iron based powders essentially free from fine particles may be obtained by removing the finer fractions of the powder or by manufacturing a powder having the desired particle size distribution. As the powders used according the present invention differ from those conventionally used one would thus expect that problems would be encountered when the components are sintered.

The influence of particle size distribution and the influence of particle shape on the compaction properties and properties of the compacted body have been subjected to intense studies. Thus the US patent 5,594,186 reveals a method of producing PM components with a density higher than 95 % of theoretical density by utilising substantially linear, acicular metal particles having a triangular cross section. Such particles are suitably produced by a machining or milling process. Powders having coarse particles are also used for the manufacture of soft magnetic components. Thus the US patent 6 309 748 discloses a ferromagnetic powder, the particles of which have a diameter size between 40 and 600 µm. In contrast to iron based powder particles according to the present invention, these powder particles are provided with a coating. In the US patent 4,190,441 a powder composition for production of sintered soft

magnetic components is disclosed. In this patent the iron powder includes particles with less than 5 % exceeding 417 μm , and less than about 20 % of the powder particles have a size less than 147 μm . This powder is mixed with specific amounts of ferrophosphorus in order to obtain the soft magnetic components.

In order to obtain compacts having satisfactory mechanical properties according to the present invention it may be necessary to add minor amounts of graphite to the powder mixture to be compacted. Thus graphite in amounts between 0.1 - 1, preferably 0.2 - 1.0 and most preferably 0.2-0.8 % by weight of the total mixture to be compacted could be added before the compaction.

Other additives which may be added to the iron-based powder before compaction such as alloying elements comprising Mn, Cu, Ni, Cr, Mo, V, Co, W, Nb, Ti, Al, P, S, and B machinability enhancing compounds and hard phase material.

The iron-base powder may also be combined with a lubricant before it is transferred to the die (internal lubrication). The lubricant is added to minimize friction between the metal powder particles and between the particles and the die during a compaction, or pressing, step. Examples of suitable lubricants are e.g. stearates, waxes, oligomers, polymers etc conventionally used. The lubricants are preferably added in the form of particles but may also be bonded to the particles. According to the present invention the amount of lubricant added to the iron-based powder may vary between 0.05 and 0.75 %, preferably between 0.1-0.6 % by weight of the mixture.

The method according to the invention may also be performed with the use of external lubrication (die wall lubrication) where the walls of the die are provided with a lubricant before the compaction is performed. A combination of external and internal lubrication may also be used.

The term "at high compaction pressure" is intended to mean at pressures of about at least 800 MPa. More interesting results are obtained with higher pressures such as pressures above 900, preferably above 1000, more preferably above 1100 MPa.

Conventional compaction at high pressures, i.e. pressures above about 800 MPa with conventionally used powders including finer particles, are generally considered unsuitable due to the high forces required in order to eject the compacts from the die, the accompanying high wear of the die and the fact that the surfaces of the components tend to be less shiny or deteriorated. By using the powders according to the present invention it has unexpectedly been found that the ejection force is reduced at high pressures, about 1000 MPa, and that components having acceptable or even perfect surfaces may be obtained.

The compaction may be performed with standard equipment, which means that the new method may be performed without expensive investments. The compaction is performed uniaxially in a single step at ambient or elevated temperature. Alternatively the compaction may be performed with the aid of a percussion machine (Model HYP 35-4 from Hydropulsor) as described in patent publication WO 02/38315.

The sintering may be performed at the temperatures normally used within the PM field, e.g. at low temperature such as 1100-1140 or higher temperatures such as 1250°C and in conventionally used atmospheres.

Other treatments of the green or sintered component may as well be applied, such as green machining, case hardening, surface densification.

In brief the advantages obtained by using the method according to the present invention are that high density green compacts can be cost effectively produced. The new method also permits production of higher components which are difficult to produce by using the conventional technique. Additionally standard compaction equipment can be used for producing high density compacts having acceptable or even perfect surface finish.

Examples of products which suitably can be manufactured by the new method are connecting rods and gears. By using stainless steel powders flanges are of special interest.

The invention is further illustrated by the following examples.

Example 1

Two different iron-based powder compositions according to the present invention were compared with a standard iron-based powder composition. All three compositions were produced with Astaloy Mo available from Höganäs AB, Sweden. 0.2 % by weight of graphite and 0.4 % by weight of a lubricant (Kenolube™) were added to the compositions. In one of the iron-based powder compositions according to the invention, particles of the Astaloy Mo with a diameter less than 45 μm were removed and in the other composition according to the invention particles of Astaloy Mo less than 212 μm were removed.

As can be seen from figure 1-1 a clear density increase at all compaction pressures is obtained with the powder having a particle size above 212 μm . From figure 1-2 it can be seen that the force needed for ejection of the compacts produced by the iron based powder composition without part particles less than 212 μm was considerably reduced compared with the ejection force needed for compacts produced from the standard iron-based powder composition. The ejection force needed for compacts produced from the iron-based powder composition according to the invention without particles less than 45 μm is also reduced in comparison with the standard powder.

A noticeable phenomenon is that the ejection force for compacts produced according to the present invention decreases with the increasing ejection pressure whereas the opposite is valid for the standard composition.

It was also observed that the compacts obtained when the standard powder was compacted at a pressure above 700 MPa had deteriorated surfaces and were accordingly not acceptable. The compacts, which were obtained when the powder essentially without particles less than 45 μm was compacted at a pressure above 700 MPa, had a less shiny surface which at least under certain circumstances is acceptable.

Example 2

Example 1 was repeated but as lubricant 0.5 % of an EBS-lubricant mixture was used and the compaction was performed with the aid of a percussion machine (Model HYP 35-4 from Hydropulsor, Sweden).

From figure 2-1 and 2-2, respectively, it can be noticed that higher green densities and lower ejection forces were obtained with the powder composition according to the invention compared with the powder composition with the standard powder. It can also be noticed that com-

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ponents produced from the standard powder had deteriorated surfaces at all compaction pressures.

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CLAIMS

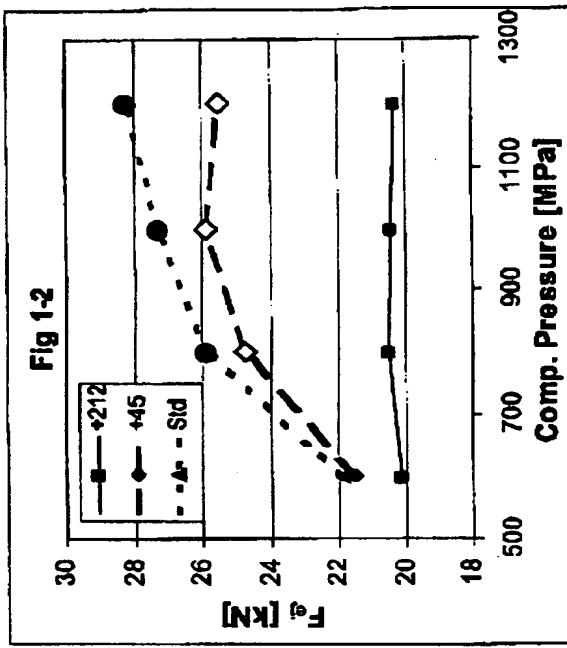
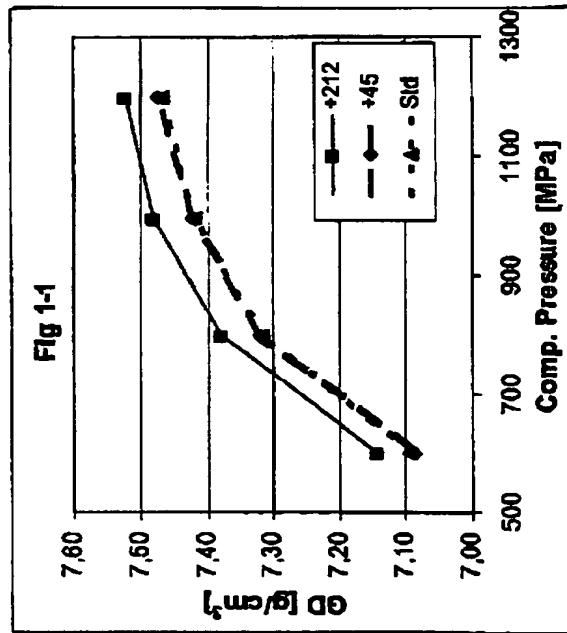
1. Process for the preparation of high density green compacts comprising the following steps:
 - providing an iron-based powder essentially free from fine particles;
 - optionally mixing said powder with graphite and other additives;
 - uniaxially compacting the powder in a die at a compaction pressure of at least about 800 MPa and
 - ejecting the green body.
2. Process according to claim 1 wherein the compaction is performed in a single step.
3. Process according to claim 1 or 2 wherein less than about 5 % of the iron-based powder particles have a size below 45 μm .
4. Process according to claim 3 wherein at least 10 %, preferably at least 40 % and most preferably at least 60 % of the iron-based powder consists of particles having a particle size above about 106 μm .
5. Process according to 4 wherein at least 10 %, preferably at least 40 % and most preferably at least 60 % of the iron-based powder consists of particles having a particle size above about 212 μm .
6. Process according to claim 5 wherein the maximum particle size is about 2 mm.
7. Process according to any of claims 2-6, wherein the graphite is present in an amount of 0.1-1.0 %.
8. Process according to any of claims 1-7 wherein the iron-based powder is combined with a lubricant in an amount between 0.05 and 0.75 % by weight before compaction.
9. Process according to any of claims 1-7 wherein the compaction is performed in a lubricated die.
10. Process according to any of claims 8-9 wherein the compaction is performed by using a combination of internal and external lubrication.
11. Process according to any of claims 1-10 wherein the additives are selected from the group consisting of alloying elements such as Mn, Cu, Ni, Cr, Mo, V, Co, W, Nb, Ti, Al, P, S and B machinability enhancing agents and hard phase materials.
12. Process according to any of claims 1-11, wherein the compaction is performed at a pressure of at least 900 MPa, more preferably at least 1000 and most preferably above 1100 MPa.
13. Process according to any of claims 1-12, wherein the compaction is performed at ambient temperature.

14. Process according to any of claims 1-12, wherein the compaction is performed at elevated temperature
15. Process according to any of claims 1-14 for preparing sintered products said process further including a single sintering step at a temperature above 1100°C.
16. Iron-based powder wherein less than about 5 % of the powder particles have a size below 45 μm .
17. Iron-based powder according to claim 16, wherein at least 10 %, preferably at least 40 % and most preferably at least 60 % of the iron-based powder have a particle size above about 106 μm .
18. Iron-based powder according to claim 17, wherein at least 10 %, preferably at least 40 % and most preferably at least 60 % of the iron-based have a particle size above about 212 μm .
19. Iron-based powder according to any of claims 16-18 wherein the particles have an irregular shape.
20. Composition comprising an iron based powder according to any one of claims 16-19 including 0.05 – 0.75 % by weight of a lubricant, 0.1-1.0 % by weight of graphite and optionally additives selected from the group consisting of alloying elements, such as Mn, Cu, Ni, Cr, Mo, V, Co, W, Nb, Ti, Al, P, S and B machinability enhancing agents and hard phase materials.

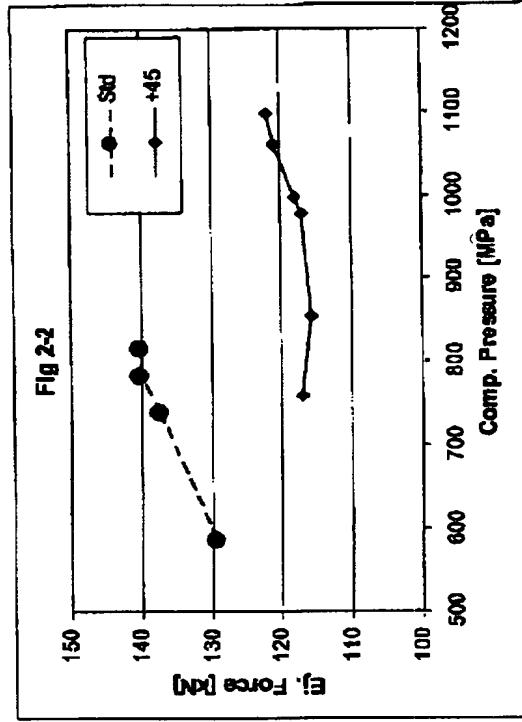
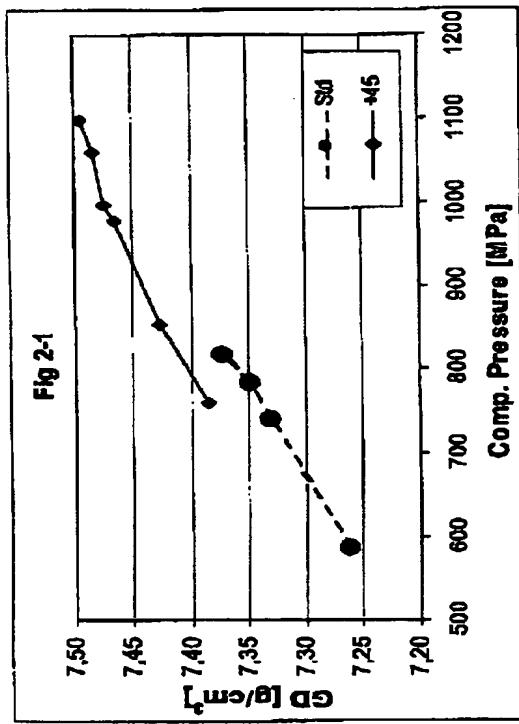
ABSTRACT

The present invention concerns a process for the preparation of high density green compacts comprising the steps of providing an iron-based powder essentially free from fine particles; optionally mixing said powder with graphite and other additives; uniaxially compacting the powder in a die at a compaction pressure of at least about 700 MPa and ejecting the green body. The invention also concerns the powder used in the method.

Example 1



- ◇ = Less shiny surface
- = Deteriorated surface

Example 2

● → Deteriorated surface